

Adjust the Magic-Angle with KBr

- (1) Spin KBr at 5 kHz
- (2) Use a one-pulse sequence with no ^1H decoupling to acquire ^{79}Br spectra. ^{79}Br is 0.36% lower in frequency than ^{13}C and has a short T_1 relaxation time, so one can pulse with a repetition rate of $<0.5\text{s}$
- (3) Put ^{79}Br peak on resonance. And using a relatively short pulse acquire a spectrum. KBr has a ^{79}Br chemical shift of 60.15ppm.
- (4) Adjust Magic-angle (MA) knob to maximize the number of observed rotational echoes observed in the FID

NOTE: *From the Bruker TopSolids Manual: "Depending on the year a standard bore (SB) probe was produced (before or after 2010), the MA adjustment screw must either always be turned clockwise or always be turned counterclockwise into the MA position (as seen from underneath the probe) in order to ensure reproducible accuracy of the MA after a sample exchange.*

In the modern design of SB probes produced after 2010, the MA screw has to be turned clockwise in order to adjust the MA with accuracy. Here you will not see a pin, but a pneumatic cylinder that is surrounded by a metallic rectangular block. The block touches the probe frame while maintaining the 'MA position', and moves away from the frame when the stator is in the 'rotor eject position'. It is important to check that the block is firmly touching the probe frame to ensure that the MA position is adopted.

If the MA screw has been turned too far, turn it back slightly and toggle the stator by a sample exchange cycle before continuing."

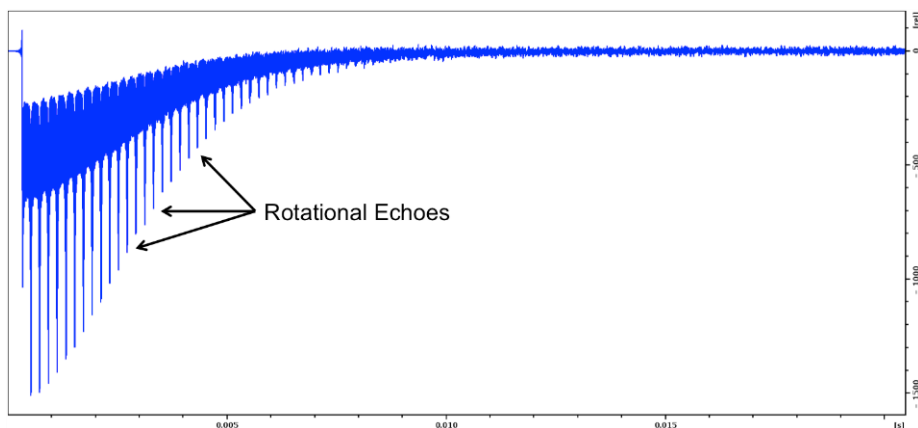


Figure 1: FID of KBr with the MA correctly set. The MA should be adjusted to maximize the number of observed rotational echoes.

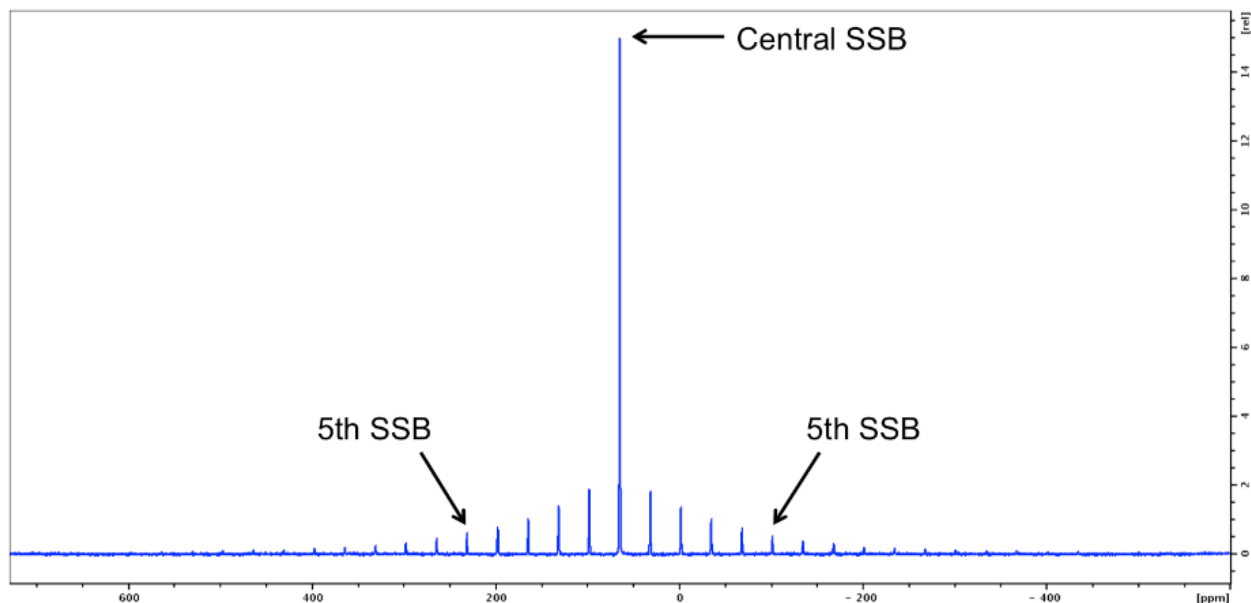


Figure 2: KBr ^{79}Br spectrum - the FWHM of the central SSB should be ~ 120 - 130 Hz and the 5th SSB FWHM should be within 10% of the measured FWHM for the central SSB.

- (5) Measure the FWHM for the central and 5th spinning side bands (SSB). The FWHM should be $\sim 120 - 130$ Hz and there should NOT be more than 10% difference between the FWHM of the central and 5th SSB.
- (6) Eject and re-insert the KBr rotor to acquire another spectrum. Ensure the FWHM of the central and 5th SSB are near the values determined in the previous step, there should not be more than a 10% change.
- (7) If the values have changed then the MA is not set properly. Re-adjust the MA and repeat until KBr can be ejected and re-inserted with a change of less than 10% between the central and 5th SSB.